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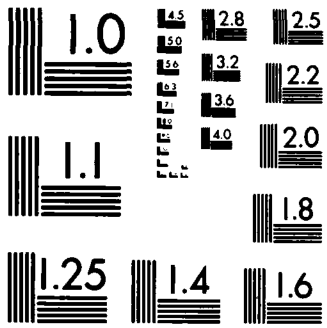

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STATIC FATIGUE OF A SINTERED SILICON NITRIDE

GEORGE D. QUINN

October 1984

Prepared under
Interagency Agreement DE-AI01-77CS51017
Task IV

by
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WATERTOWN, MASSACHUSETTS 02172-0001

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

The static fatigue resistance of a sintered silicon nitride was assessed at elevated temperature in air. The material was developed for heat engine applications. No difference in ambient or elevated temperature behavior was noted for the injection-molded or slip-cast versions. The material can maintain 150 MPa flexural stress for up to 1000 hours at 1200°C but is susceptible to slow crack growth and creep phenomena at higher stresses. Originator-supplied keywords include:

A-1

INTRODUCTION

Sintered silicon nitride holds promise for applications in new high performance engines. This report presents strength and static fatigue results for a new form of sintered silicon nitride suitable for such applications. Similar testing has been previously performed and presented for a wide range of high performance ceramics.[1-4]

MATERIAL

The sintered silicon nitride tested is a commerically available material containing 6 w/o Y_2O_3 and 2 w/o Al_2O_3 sintering aids.* This material has undergone processing development resulting in high strength and good oxidation resistance.[5-8] Two methods of near-net shape fabrication were used to prepare the material.

Slip casting was used to produce disk samples with sintered dimensions approximately 7.0 cm diameter and 0.95 cm high. Two disks were produced which had sintered densities of 3.23 g/cm^3 as determined by the Archemedes method. Injection molding was used to fabricate 35 bars of size 0.39 x 0.74 x 5.6 cm that had densities between 3.20 g/cm^3 and 3.23 g/cm^3. Both materials were sintered above 1800°C in a nitrogen atmosphere. Flexure specimens measuring 0.22 x 0.28 x 5.1 cm were ground with a 320 grit final longitudinal surface finish and chamfered along the edges. The geometric density of both slip-cast and injection-molded bars averaged 3.23 g/cm^3 with a standard deviation of 0.01 g/cm^3. Polished sections of both injection-molded and slip-cast forms revealed that the material was fully dense with negligible residual porosity. X-ray diffraction discerned β Si_3N_4 and a minor second phase which could not be identified.

EXPERIMENTAL PROCEDURE

The room temperature (RT) flexure strength was determined to serve as a reference. Four-point flexure was used with spans of 2.03 cm x 4.06 cm and a crosshead rate of 0.13 cm/min. Relative humidity was 20% to 35%. Sixteen specimens, each of injection-molded and slip-cast materials, were tested.

*GTE AY-6 Grade, GTE Laboratories, Waltham, Massachusetts.

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Elevated temperature testing in air began with stepped temperature stress rupture (STSR) experiments[1,9] which were intended to discern any usual temperature problems in the 800°C to 1225°C temperature range. Additional isothermal stress rupture experiments were done at 1200°C which is a temperature common to earlier studies.[1-4] Flexural fixtures had spans of 1.91 cm x 3.81 cm. Applied stresses were computed using the elastic formulation.* Additional details of the experimental procedure are in Reference 1. Permanent tensile strains at the surface (0.05% to 2%) were evaluate by photographically enlarging the specimen curvature and measuring the midspan deflection relative to the inner load bearing points.

RESULTS

The room temperature strengths of the injection-molded and slip-cast specimens are similar as shown in Table 1 and Figure 1. The Weibull parameters were evaluated by a least squares fit to a two-parameter function.† The characteristic strength indicated is the stress corresponding to the 63 percent failure probability of the flexure specimens. The strength distribution differences are not statistically significant given the scatter in the data. The strengths are somewhat below those reported in Reference 8, but this is not surprising since the latter were generated with smaller specimens in three-point flexure. The most common strength-limiting defects were pores or porous zones, often having a planar aspect suggestive of a seam or crack. Occasionally, iron inclusions or possibly machining damage (the latter in the very high strength specimens) were strength limiting. Similar defects have been previously reported in this material.[8]

Table 1. ROOM TEMPERATURE FLEXURAL STRENGTH (MPa)

	Average Strength	Standard Deviation	Weibull Modulus	Flexure Specimen Characteristic Strength
Slip	651	109	5.8	702
Injection Molded	634	86	7.5	675

Figure 2 illustrates the STSR sequence used for this study. The specimens were loaded and maintained at 800°C for twenty-four hours. The temperature was then increased as shown, with twenty-four hour holds at each step. Each labelled arrow designates one experiment; the arrow indicates the time of failure (or survival after 1225°C), and the number gives the applied elastic stress in MPa. Eleven slip-cast and nine injection-molded specimens were tested. The results show that there is no significant difference between the slip-cast and injection-molded material. Intergranular crack growth zones were evident on the fracture surfaces of the specimens which failed at 1100°C and 1225°C. The specimens which reached 1225°C had significant permanent creep strains (of the order of tenths of percent) indicating a creep fracture regime. At lower temperatures (800°C to 900°C), optical microscopy revealed that there were no prominent crack growth zones. Failures were preferentially from surface-connected porous zones, similar to those observed in the room temperature reference specimens. None of the specimens showed evidence of catastrophic oxidation reactions characteristic of some silicon nitrides fabricated with yttria additives.

*$\sigma = Mc/I$ where σ is stress, M is the applied moment, I is the moment of inertia, and c is the half-height of the specimen.

†The ranking probability used was $i/N+1$, where i is the i^{th} datum and N is the total number of specimens.

9. QUINN, G. D., and KATZ, R. N. *Stepped Temperature Stress-Rupture Testing of Silicon Based Ceramics.* Am. Ceram. Soc. Bull., v. 51, no. 11, 1978, p. 1057.

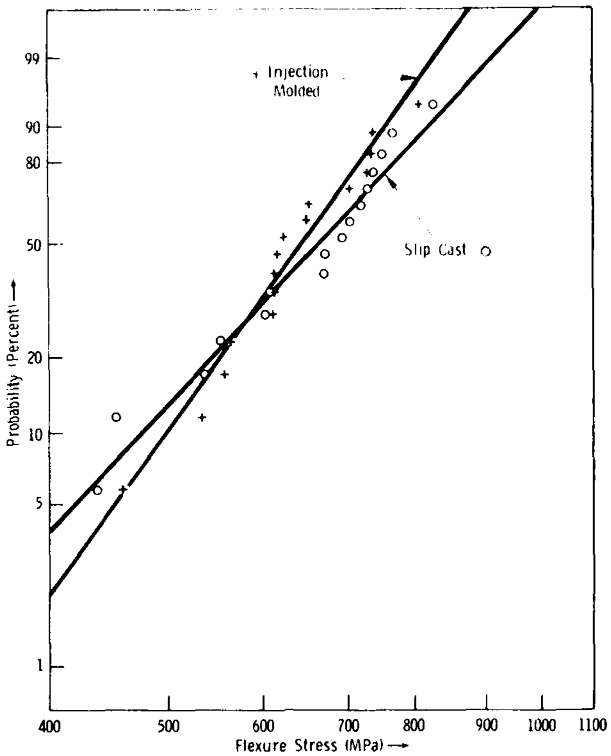

Figure 1. Weibull plot of the room temperature strength of sintered silicon nitride.

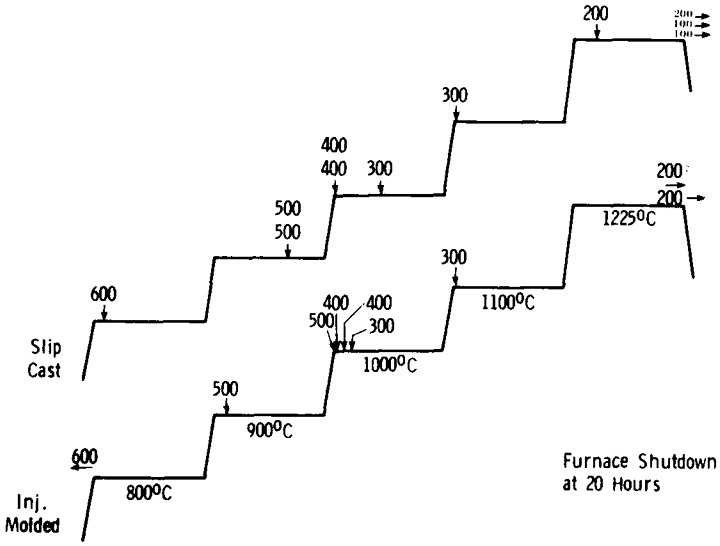

Figure 2. Stepped temperature stress rupture (STSR) results.

Both slow crack growth from preexisting flaws and creep fracture mechanisms of static fatigue were detected by the 1200°C isothermal stress rupture experiments shown in Figure 3. The observed crack growth patterns are believed to fall into two categories dependent on the applied stress. Under high stresses (greater than 300 MPa), times-to-failure and creep strains were small (≤ 0.2%). Intergranular crack growth zones were observed centered upon the same flaw type which were strength limiting in the room temperature specimens (Figure 4). Figure 3 suggests the fast fracture strength at 1200°C would be about 400 MPa, which is comparable to earlier published data.[8]

Specimens under lower stress (150 MPa to 300 MPa) had considerable creep strain (~ 0.4%) after 10 hours and strains as high as 1.5% to 2.0% after 1000 hours. These specimens showed numerous creep cracks on the tensile surface (Figure 5), some extending deeper than half the specimen thickness (Figure 6). Such creep cracks are often associated with cavitation due to grain boundary sliding which may not necessarily involve preexisting flaws.

Similar observations showing the presence of both slow crack growth from preexisting flaws and creep fracture have been presented for hot-pressed silicon nitride[*,10] and for sintered silicon nitride.[11]

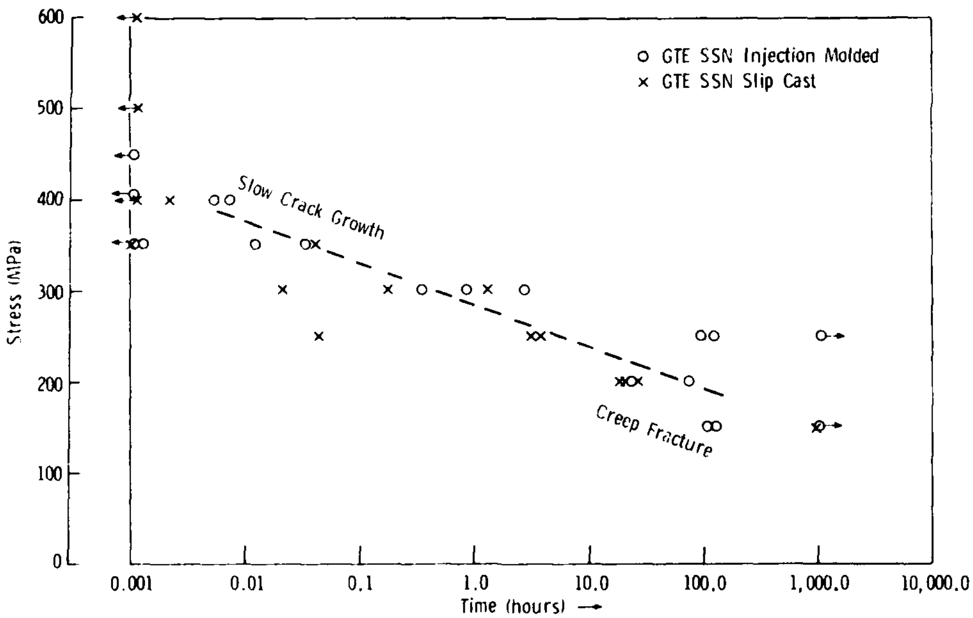

Figure 3. Stress rupture at 1200°C.

*QUINN, G. D. *Fracture Mechanism Map for Hot Pressed Silicon Nitride.* To be published in Ceramic Proceeding, 1984.
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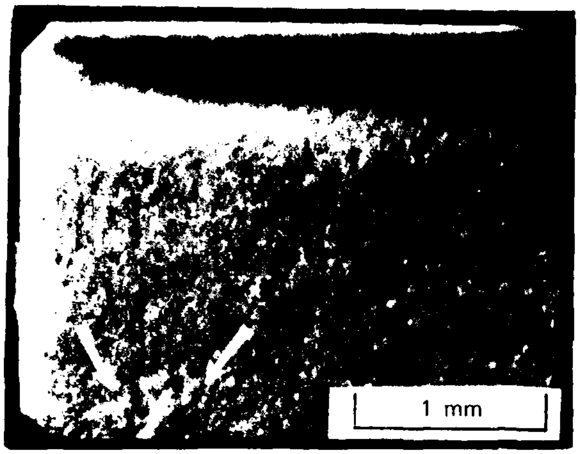
11. ODA, I., MATSUI, M., and SOMA, T. *High Temperature Fatigue in Pressureless Sintered Nitride.* Progress in Nitrogen Ceramics, F. L. Riley, ed., Martinum-Nijhoff Publishing Company, Boston, Massachusetts, 1983, p. 501-506.



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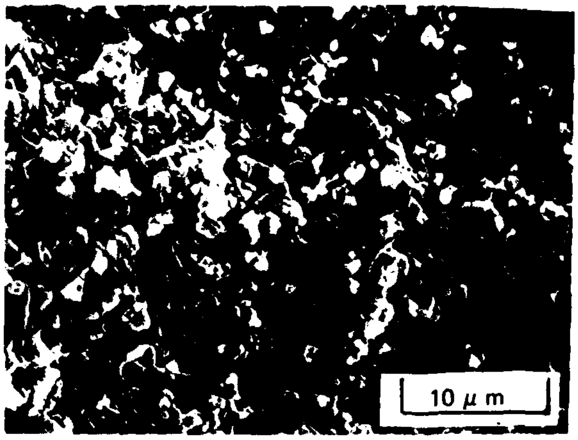

(b)

Figure 4. [redacted] a) Fracture surface of a specimen that failed in 0.2 hours at 1200°C. The arrows point out a slow crack growth zone. Creep strain was 0.1%. b) Close-up of the SCG zone showing that it is intergranular.

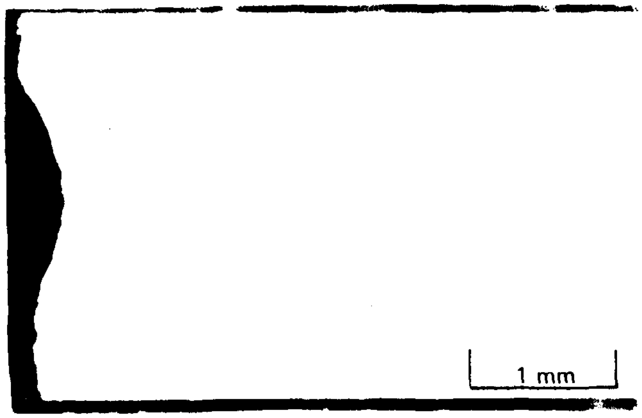

Figure 5. Tensile surface of a specimen that failed after 76 hours at 1200°C with a 200 MPa applied stress. Numerous creep cracks are evident. The creep strain was 0.8%.

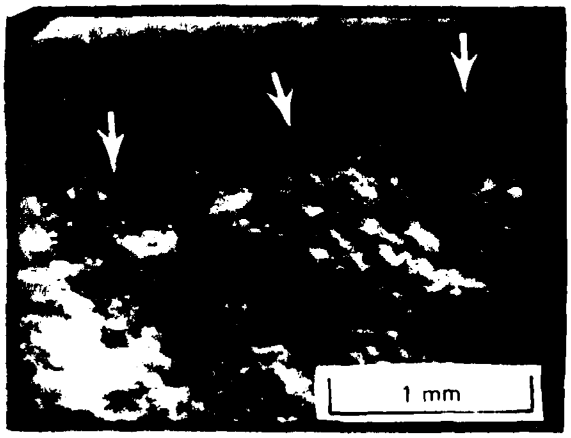

Figure 6. Fracture surface of a specimen that failed at 108 hours at 1200°C. The applied stress was 150 MPa and the final creep strain was 0.8%. The arrows delineate the creep crack growth zone.

CONCLUSION

The slip-cast and injection-molded sintered silicon nitride demonstrate similar mechanical behavior at ambient and elevated temperature. Crack growth zones were evident on specimens tested at 1000°C and above.* The observed creep and crack growth behavior are intergranular which undoubtedly involves the grain boundary phase.

At 1200°C and under high stress (>300 MPa), crack growth is initiated from preexisting flaws with short times-to-failure and small creep strains. Surface-connected porous zones may be especially vulnerable sites for static fatigue phenomena. Under lower stress (150 MPa to 300 MPa), specimens have longer times-to-failure, higher creep strains (>0.4%), and exhibit multiple noncatastrophic cracks on the tensile surface. This extensive creep cracking is characteristic of a creep fracture mode of static fatigue.

This sintered silicon nitride can maintain a reasonable load-bearing capability, up to 1200°C for long durations, but this is accompanied by substantial slow crack growth and creep. Further processing work on this system may lead to improved grain boundary phase refractoriness and, thus, improved creep and static fatigue resistance. Lowering the alumina content is one method of improving the high temperature properties.[8] Preliminary findings indicate that a silicon nitride fabricated with 6 w/o Y_2O_3 and no alumina had vastly superior elevated temperature strength, crack growth, and creep resistance.[8,12] However, a trade-off in fabricability occurs for such compositions.

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*Subsequent to the writing of this report, nineteen additional specimens were tested in stress rupture at 1000°C. Eleven time dependent failures occurred due to slow crack growth at stresses from 400 to 600 MPa. The nineteen experiments suggested a fatigue curve similar to Figure 3, but shifted to higher stresses.

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